

USES OF CAUSTIC SODA RECOVERED FROM THE MERCERIZATION  
PROCESS IN THE TEXTILE INDUSTRY

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Douglas Franklin Becknell

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of the Requirements for the Degree

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PROCESS IN THE TEXTILE INDUSTRY

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Dedicated to

MY PARENTS

MR. AND MRS. WALLACE W. BECKNELL, JR.

For Their Encouragement

and to

DR. WILLIAM L. HYDEN

For His Guidance

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## SUMMARY

Every year thousands of dollars worth of chemicals used in processing solutions are discharged into the effluents from industrial facilities. This figure represents a major economic waste. In addition, some solutions are detrimental to the quality of water of the streams into which they are discharged since this diluted process waste must be purified at considerable expense before it can be used again.

This investigation concerned the satisfactory reuse of sodium hydroxide, which is widely used in the wet processing of cotton textiles. Because such a large quantity of caustic soda is used in the mercerization process, it was selected as the primary source from which the caustic was recovered. Emphasis was focused on low cost inside-the-plant modifications for recovery to greatly reduce the quantity which enters the effluents. Means were developed by which the relatively low caustic concentration from the wash boxes following mercerization could be considerably increased.

The recovered caustic soda was reused in selected textile processes of desizing, scouring, and mercerizing under plant and laboratory conditions. The mercerizing process was found to be suitable for the use of the major portion of the recovered caustic. In addition, the recovered caustic was suitable for the preparation of hypochlorite bleach.

The economics and feasibility of the recovery and reuse of caustic soda are discussed. The conclusion is reached that caustic soda can be recovered and reused satisfactorily, and that in so doing, textile plants

may increase profits and reduce stream pollution.

Because product quality is a major criterion by which all goods are sold, special emphasis was placed upon the quality of the textile products made using recovered caustic.

The recommendations of this study contain proposals for further studies with the aim of avoiding pollution through more efficient use of raw materials. The ultimate aim is for an entirely closed system to conserve water and chemicals.

## CHAPTER I

### INTRODUCTION

#### Purpose of the Research

The purpose of this research was to demonstrate, in a textile mill, how to purify caustic soda recovered from the mercerizing process by simple means and to reuse it in various wet processes. This study is primarily oriented toward the smaller mills because a large number of these do not have sufficient volume of production to make recovery and reconcentration by evaporation feasible.

#### In-Plant Textile Effluent Handling

##### Reuse Possibilities

Today, there is considerable pressure being exerted, by both federal and state governments, on industries to avoid pollution of the streams. The textile industry is involved because, as one of the oldest and largest important enterprises in this country, the effluents from the wet processing operations can and do pollute streams unless means of treatment are applied. The large textile mills frequently have means for waste treatment and for recovery of ingredients from the wash water and effluents. The small mills, on the other hand, may not have funds available nor does volume of production justify capital outlays necessary to recover and reuse materials from the effluents. Thus, means of meeting the problems which cause this dilemma for the smaller mills are needed. Reuse of caustic soda from the wet processing of textile, if recover-

able in suitable quality, is an excellent important step toward solving the problem. This is particularly so because caustic soda is widely used, represents considerable processing chemical cost for cotton finishing, and is an undesirable pollutant.

#### Possible Sources of Sodium Hydroxide

Because caustic soda is used in the processes of desizing, scouring, bleaching, mercerizing, and dyeing, all these operations present possible sources from which to recover caustic solutions. However, mercerization uses much larger quantities of caustic soda than the other processes and is therefore the most promising operation for study if one wishes to make a major reduction in caustic discarded.

#### Effect on Pollution

Some plants discharge raw waste directly into the streams; others dispose of their waste through the city sewerage system. For these plants which have no waste disposal treatment facilities and have no city sewerage systems available, this present work may be of particular interest. However, all mills are concerned with keeping caustic concentration in the effluent to a minimum. For this reason, this investigation was focused on inside-the-plant modifications to reduce the quantity of caustic soda discharged into the effluent and may therefore be of industry-wide interest. By altering in-plant processes and decreasing the quantity of effluent, pollution will be reduced.

The plant at which this study was made consumed more than 400 tons of caustic soda annually. The cost for 50 per cent caustic soda was approximately \$71 per ton. It is axiomatic that the reuse of all available caustic soda at the lowest investment is most attractive. As pointed out

by Jones (1) a plant must have a certain minimum volume to justify the recovery of caustic by means of dialysis and evaporation. Those plants which do not have sufficient volume to justify such expenditures still need to have a means for keeping the concentration of caustic soda discharged into the streams at the absolute minimum.

#### Balancing of Effluent

Recovery of caustic soda from desizing, scouring, bleaching, and dyeing presents many complex problems which are beyond the scope of this immediate investigation. Because of the nature of the desizing and scouring processes, the recovery of caustic soda solutions of satisfactory quality from these present major complicated problems. Bleaching and dyeing likewise offer little opportunity for purification and reconcentration by simple means. However, this is not to imply that the effluents from these processes should be discarded directly into the stream. While such effluents are contaminated they are diluted and may indeed find important use in neutralizing acidic effluents from other wet finishing operations and thereby aid in increasing pH of the total effluent. Acids are frequently used in dilute concentrations in textile finishing. The contaminated and dilute caustic soda from desizing, scouring, and dyeing can perhaps be used to real advantage to neutralize acidic effluents.

#### Importance of Caustic Soda in Wet Processing of Cotton

Free caustic soda is never left in the fabric after mercerization but is completely washed out. In some mills the wash water is then discarded as a part of the effluent. If a means can be developed to recover the caustic soda in a quality satisfactory for reuse, the plants will bene-

fit from savings of caustic soda while desirably reducing the polluting effect of the high alkalinity. Recovered wash waters containing 6 to 8 per cent sodium hydroxide has important reuse values. By diluting such wash waters with fresh water, the caustic soda solution may be used in desizing and scouring. Furthermore, if this 6 to 8 per cent caustic solution is of satisfactory quality for diluting the 50 per cent technical grade caustic to a concentration of approximately 23 per cent, it can be reused for mercerization with satisfactory results and with economic advantages. Other possibilities for the caustic wash water solutions are for the manufacture of hypochlorite bleach and for pH control in bleaching and dyeing operations.

#### Summary of Literature and Plant Surveys

Relatively little information has been published concerning the recovery and reconcentration of caustic soda from the wash water of the mercerizer aside from that which concerns dialyzers and evaporators. Wilkerson and Rochelle (2) discuss how an evaporator has affected their plant. Sanders (3) suggests the removal of colloidal impurities from caustic soda solutions by the addition of sodium carbonate and calcium hydroxide followed by boiling and decanting of the clear solution. Panizzon (4) proposes the elimination of organic matter from the mercerization wash water by use of the ultra centrifuge, dialysis, and evaporation. The writer obtained considerable information on practices used in Georgia mills by visiting 48 textile wet processing plants, many of which used mercerization as one of their finishing processes. Procedures at the mills visited varied in recovery practices from none at all to com-



plete recovery using triple effect evaporators.

### Economics and Feasibility of Recovery

To determine if it is feasible to recover and reconcentrate caustic soda using evaporators, two important factors must be established. First, the volume of caustic soda to be reconcentrated, and second, the desired concentration must be estimated and measured. If the volume of a mercerizing plant is sufficiently large, recovery by the use of evaporators is attractive. According to Jones' earlier work (5) recovery of 400 tons of caustic soda annually would cost \$12.50, \$23.80, and \$27.50 per ton using a single, double, and triple effect evaporators reconcentrating to 20, 34, and 45 per cent, respectively. This is to be compared to the commercial rate of \$71 per ton delivered plus freight and handling. The break-even points for the three levels of evaporation in the plant he studied were 71, 134, and 155 tons of caustic soda per year. Evaporators are quite feasible for plants with volumes larger than their break-even point. Adversely, to a plant using smaller quantities, they are not economical. This investigation shows that there are avenues other than evaporators for recovery and reuse of caustic soda which are practical, particularly for the small plant. Incidentally, it is also applicable to medium and larger plants. The scheme employs a relatively simple low cost system which permits reuse after the caustic wash water has been filtered, collected in a storage tank, and used as needed as the diluent for 50 per cent technical grade caustic soda. The experimental work done to demonstrate this hypothesis is discussed in subsequent sections of this thesis.

## CHAPTER II

### EXPERIMENTAL PHASE

#### Scope of the Investigation

This research concerns the reuse of caustic soda collected from the wash water following mercerization. Specifically, the investigation was focused upon ways and means for collection, storage, purification, and reuse of caustic soda in textile operations. The plan of attack and steps taken were as follows:

- a. A complete analysis was made of the recovered caustic soda including the suspended solids and the dissolved materials.
- b. A relatively simple system was designed for the removal of extraneous material from the recovered caustic soda.
- c. Laboratory and plant scale experiments were conducted to establish the effectiveness of the recovered caustic soda in selected textile operations.
- d. A careful comparison was made of the quality of the textile products produced using the recovered caustic soda with those products made utilizing technical grade caustic soda.

A local plant which was not recovering caustic soda from wash waters co-operated in this investigation. The mercerizing and washing system at this plant was similar to that generally used in the textile industry when caustic soda is not recovered. The cloth mercerized was 100 per cent

cotton. In these experiments, the caustic samples were taken only when fabrics contained no dyed or colored yarn.

#### Collecting the Caustic Soda

The wash water immediately following the mercerizer is the most important source for recovering caustic water. In industrial installations wash systems utilizing the principle of cross-counter-current-flow has been found to be highly efficient and is the preferred washing system for the fabric. However, the plant cooperating in this investigation used a series of wash boxes with each having an individual supply of fresh water. Thus, the wash system available did not have the advantage of the concentrated effluent possible with counter-current-flow. Because of this situation, some method of reconcentrating the caustic water was very desirable. The actual composition of caustic soda solution in the cloth before entering the wash boxes was approximately 21 per cent. Jones showed in his study that 65 per cent of the caustic soda was removed from the fabric by the first wash box. Therefore, this is the logical location for the collection of the caustic solution.

Even with 65 per cent of the caustic in the fabric contained in the first wash box, the volume of water passing through this box gave a caustic concentration of only 2 to 3 per cent, which is too low for feasible reuse. It was found that by reducing the rate of flow of water through this first wash box a considerable increase in the caustic concentration could be achieved without causing a significant increase in concentration in the last wash box in the series, see Table 1. Also, there was no increase in the sodium hydroxide in the delivered cloth.

Table 1. Results of Change in Flow of Water Through Buffer Box

Sample	Per Cent Caustic in Buffer Box	Per Cent Caustic in Last Wash Box
1	2.68	0.24
	5.64	0.24
2	3.44	0.28
	6.72	0.28
3	3.97	0.05
	5.44	0.06
4	4.28	0.23
	6.00	0.23
5	3.84	0.16
	5.68	0.17
6	5.76	0.20
	6.96	0.20
7	3.76	0.25
	5.32	0.26

This was important because the cloth must be as caustic-free as possible. This reduction in the flow of water created an intermediate or "buffer" zone and permitted the concentration of caustic soda to be raised from 2 to 3 per cent to 6 to 8 per cent depending upon the weight of the cloth being mercerized and the rate at which it is mercerized. This wash box will henceforth in this dissertation be referred to as the "buffer" box.

#### Present Equipment Arrangement

The complete treatment of a typical fabric through the wet processing at the plant where this study was made is as follows. First the fabric is singed using gas flames to burn off the fiber ends from the surface. This is followed by desizing with enzymes and scouring with a caustic bath in a progressive jig. Bleaching, the next step, is done continuously in open width using hydrogen peroxide. The cloth is then mercerized with caustic soda of strength 24 degrees Baumé. All goods were dried before processing, and the mercerizing bath was padded-on using the dip-squeeze-dip-squeeze method. The cloth traversed 50 feet under controlled width and length on a tenter frame. The caustic soda is in contact with the cloth from the first dip until it leaves the tenter frame and enters the washing operation. The time in the tenter varies somewhat upon the product being made, but in most instances, was approximately 37 seconds. After leaving the tenter frame the fabric entered a series of wash boxes each of which had a continuous supply of fresh water. The caustic soda removed during washing was discarded into the sewer. Following washing the fabric was folded into a box, and in many instances a second washing was given to insure the removal of any residual caustic before drying.

If desired, the fabric was now dyed.

#### Analysis of the Recovered Caustic Soda

An analysis of the caustic wash water was made to determine if any harmful impurities were present. This was necessary because some impurities such as pectins can cause unfavorable results in shade matching when dyeing the fabric. The organic matter of the wash water was separated using carbon tetrachloride as the solvent. A drying agent, anhydrous magnesium sulfate, was used to remove any water from the carbon tetrachloride extract. The organic portion of the sample was then separated by distillation into four fractions. A Perkin-Elmer 221 Spectrophotometer was used to determine the characteristic absorption spectra of the extracted material. The spectra observed were compared to those of known substances. The drying of the unknown was essential because sodium chloride cells were used as containers. A standard cell containing pure solvent was placed in the reference beam to balance the absorption spectra of the unknown carbon tetrachloride extract. A graphical recording was made of each of the four fractions over the entire infrared region.

The inorganic portion of the caustic solution was analyzed using standard procedures as outlined in Introduction to Semi-Micro Qualitative Chemical Analysis (6).

#### Removal of Extraneous Material

According to Edelstein (7) the presence of large quantities of impurities such as sodium carbonate and pectins cause a decrease in both luster and dye affinity of fabrics. Most of the fabric to be mercerized had also been singed, desized, scoured, and bleached, thus removing many

impurities from the fabric. In many instances the impurities in the recovered wash water were as low as 4.5 parts per thousand, but when fabrics were mercerized in the greige, impurities were as high as 19.5 parts per thousand. Because of this large range of solids present, all the caustic wash water should be filtered. Commercial filters are available with capacities large enough for any volume that may be encountered. One excellent type of filter is a vibro-screen separator with a 30-inch diameter and a 150 mesh stainless steel screen. It is capable of handling up to 60 gallons of solution per minute. The f.o.b. cost of this separator is less than \$2,000. A wide range of capacities may be obtained using a variety of combinations of diameter with different mesh screens.

The caustic solution from the "buffer" box should be collected in separate storage tanks. Otherwise, the benefits of the "buffer" box will be lost. Because of this requirement, two separators of the type described above will be needed. The caustic solution from each separator will be directed to the proper location and reused by being adjusted to the proper strength using a 50 per cent stock solution of caustic soda.

#### Use of Caustic Soda in Selected Operations

Many of the experiments were conducted in the plant under normal operating conditions. In others, plant conditions were simulated in the laboratory. The areas selected for the reuse of the caustic solution and the procedures followed are discussed in the following paragraphs.

#### Desizing and Scouring

Enzymes in alkaline medium were used in the plant for desizing. Caustic soda is also used in the scouring operation. The purpose of

these processes are to remove the size that was applied to the warp prior to weaving and to remove other non-lint material. The evaluation of these processes includes the determination of the total per cent non-lint material removed.

In the laboratory a scale model automatic jig, to simulate the progressive jig in the plant, was used. Caustic soda concentrations of 2, 4, and 6 per cent were used at a temperature of 95°C for 45 minutes. The jig was then drained and the cloth was given two ten-minute rinsings with water at 95°C.

To calculate the per cent non-lint removed, the samples were first dried in an oven at a temperature of 225°F for 24 hours. The samples were then weighed and the results recorded. The samples were then desized and scoured using caustic concentrations of 2, 4, and 6 per cent. For enzyme desizing a commercially available product was used. After washing, the samples were again dried in an oven for the same length of time and at the same temperature as before. The equation used to calculate the non-lint removed was as follows:

$$\text{Per Cent N L} = 100 \times \frac{D1 - D2}{D1}$$

where,

N L = Non-lint removed

D1 = Bone dry weight before desizing

D2 = Bone dry weight after desizing

### Mercerizing

The mercerization process is one in which a fabric, usually cotton



or a cotton blend, is passed through a caustic soda solution of 17 to 23 per cent. "This causes a change in the absorptive capacity and chemical reactivity due to changes in internal structure of the cellulose--the change from native to hydrate variety." (8) "The mercerization of cotton yarn and cloth is carried out mainly to increase dye affinity and luster of the material." (9) These two criteria will thus be used to evaluate the results of the mercerization process.

Because processes which improve luster may reduce dye affinity, a decision must be made as to which is the more desirable and conditions set to obtain the optimum. If both are desired a compromise is necessary. The tension applied to the cloth as it traverses the tenter frame is an important factor for controlling both luster and dye affinity. The greater the tension the greater the luster, and the lower the dye affinity.

In the plant the experiments were performed using normal operating conditions. The caustic solution was collected from the "buffer" box in a 200-gallon tank. The concentration of the caustic solution in this tank was fortified to 24 degrees Baumé using 50 per cent caustic soda. A wetting agent was then added, and the caustic was pumped into the saturator. Two hundred to three hundred yards of cloth were mercerized and washed using the recovered caustic and an equal quantity using the technical grade caustic. Samples were taken from fabric mercerized with technical grade caustic and the reused caustic. These fabrics were then dyed in the plant using normal operating conditions and appropriate samples were taken.

"Luster is purely a surface property, the reflection of light from

a smooth surface; the smoother the surface the greater the luster. Therefore, when cotton is mercerized under tension, its surface must be rendered smoother." (10) A light ray striking a lustrous surface is partly reflected as from a mirror and partly diffused as from a matt surface. To compare the luster of the fabric a photoelectric cell was used to measure the reflectance. A diagram of the instrument is shown in Figure 1. A 6-volt battery was used to provide current for the light source, and a 4.5-volt battery provided current for the sweeping action of the X, Y recorder which gave a graphical reproduction of the reflectance. A dark room was always used because of the high sensitivity of the cell, and the samples were tested in both the warp and filling direction to give a true indication of the luster. The light source was perpendicular to a moveable table that traversed a distance of three inches; the photoelectric cell was at a 30-degree angle with the table. The average of the reflectance for warp and filling was calculated from the graphs and compared for each sample.

To evaluate the dye adsorption, each of the dyed samples was viewed by a color-eye colorimeter/abridged spectrophotometer from which points were obtained to plot a spectrophotometric curve. Some people have the ability to detect a 5 per cent difference in reflection with the naked eye, but very few can detect a 3 per cent variation (11). The spectrophotometric curves offer a quantitative measure of the reflectance of light of different wave lengths from which the differences between fabrics can be evaluated.

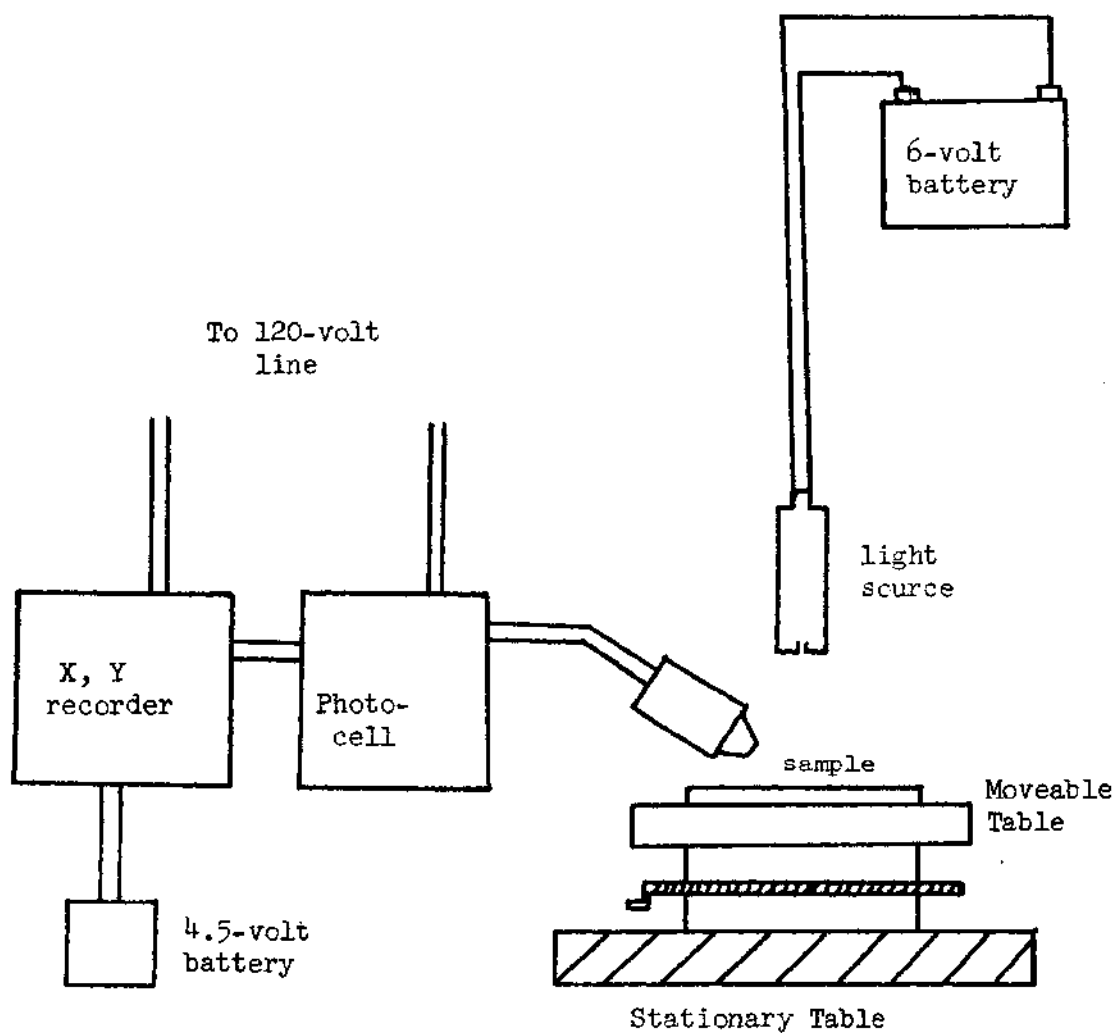


Figure 1. Instrument for Measuring Luster

### Mercerization as a Variable in Dye Adsorption

An experimental program was conducted in the laboratory for the purpose of determining the most desirable conditions for mercerization to obtain maximum dye adsorption. Two variables were employed; one, the per cent caustic soda in the mercerizing bath, and two, the contact time between the caustic and the fabric. Caustic concentrations of 18, 20, and 23 per cent were used with a constant time of 75 seconds for the first series, and exposure times of 15, 30, 45, 60, and 105 seconds were used with a constant concentration of 22.5 per cent for the second series. The same color-eye colorimeter/abridged spectrophotometer as above was used to evaluate the results in terms of per cent absorbency. The smaller the reflectance of a sample the greater the quantity of dye absorbed. By increasing the absorbency of the fabric to its maximum through optimum mercerization, lesser quantities of dyestuffs may be used.

### Bleach Preparation

This experiment was also conducted in the laboratory using recovered caustic soda and chlorine gas. Theoretically, full saturation is achieved between 40 weight units of caustic soda and 35.5 weight units of chlorine. An excess of caustic soda is usually available to prevent the formation of free hypochlorous acid, which causes an unstable bleach solution.

The bleach was prepared by bubbling chlorine gas slowly through a caustic soda solution. Chlorine was added at a controlled rate, and dispersion tubes were used to assure complete absorption. The bleach solution was analyzed by determining the strength of the solution in grams per liter of available chlorine and by the stability of the solution.

The strength of the bleach was determined using a standard sodium thiosulfate titration. To determine stability the bleach solution was set aside for five days and then titrated again; the decrease in grams per liter of available chlorine was noted. A comparison of the reflectance of two fabrics bleached with equal strength solutions of a commercial bleach and the experimental bleach was also used in the evaluation. The same photoelectric cell used in luster determinations was used to measure the difference in brightness of the two fabrics.

### CHAPTER III

#### DISCUSSION OF RESULTS

The results from this research, in cases where the fabric was the criterion, were evaluated using subjective methods rather than by absolute standards. This was necessary because the cooperating company does commercial finishing with a wide variety of cloth to process, and the customer is the final judge as to the acceptability of a fabric. Therefore, the samples considered acceptable by the customers were taken as standards. This means that each fabric and each color has a separate standard.

#### Analysis of the Recovered Caustic Soda Solution

The purpose of this analysis was to determine what contaminants were present in the caustic wash water. The insoluble solid matter in the recovered caustic was first studied. These consisted primarily of lint from the surface of the cloth. Other components were pieces of seeds, leaves, and stems from the cotton plant.

Some dissolved solids can cause great difficulty because their presence has pronounced effects on shade matching in dyeing. An analysis of the water supply to the plant was conducted by the United States Geological Survey and is recorded in Table 2. An analysis of the caustic wash water revealed the presence of sodium, calcium, and iron. These were usually present as carbonates, sulfates, and nitrates. Thus, the carbonate ion was the only one added that was not originally present.

Table 2. Analysis of Water Supply After Treatment

Ions Present	Quantity in Parts per Million
Silica	19.00
Iron	.03
Manganese	.00
Calcium	11.00
Magnesium	1.80
Sodium	6.10
Potassium	.00
Carbonate	.00
Bicarbonate	36.00
Sulphate	9.80
Chloride	5.40
Fluoride	.95
Nitrate	.10

The analysis of the infrared spectra of the organic portion of the caustic wash water indicated that the contaminants present were likely a mixture of high molecular weight hydrocarbons. The presence of the long chain aliphatic substance was evident by the lack of absorption in the aromatic region.

The infrared spectra contained absorption in the following areas: A strong and clearly defined peak occurred at 3.4 microns. A weaker but clearly defined peak occurred at 3.5 microns with weak and broad peaks at 6.8 and 7.25 microns. Absorption in the 3.3 to 3.5 micron region was attributed to C-H stretching; whereas, C-H bending resulted in absorption at 6.7 and 7.25 microns. These spectra had absorption at the same wavelengths as the spectrum of nujol, which is a mixture of high molecular weight hydrocarbons. Samples of the spectra are reproduced in Figures 2 and 3.

#### Desizing and Scouring

Three different fabrics were selected for desizing and scouring treatments. Table 3 gives the results obtained from desizing and scouring using technical grade caustic and recovered caustic. Two per cent enzyme, commonly used throughout industry, was used in all desizing experiments. The caustic soda concentration was varied between 2 to 6 per cent. As is seen in Table 3, there is little difference in non-lint removed between samples using technical grade and recovered caustic, but the recovered caustic gave slightly better results. The non-lint removed, which may also be called non-cotton, is a measure of the amount of size, leaf, seed, and any other foreign matter removed during the desizing and



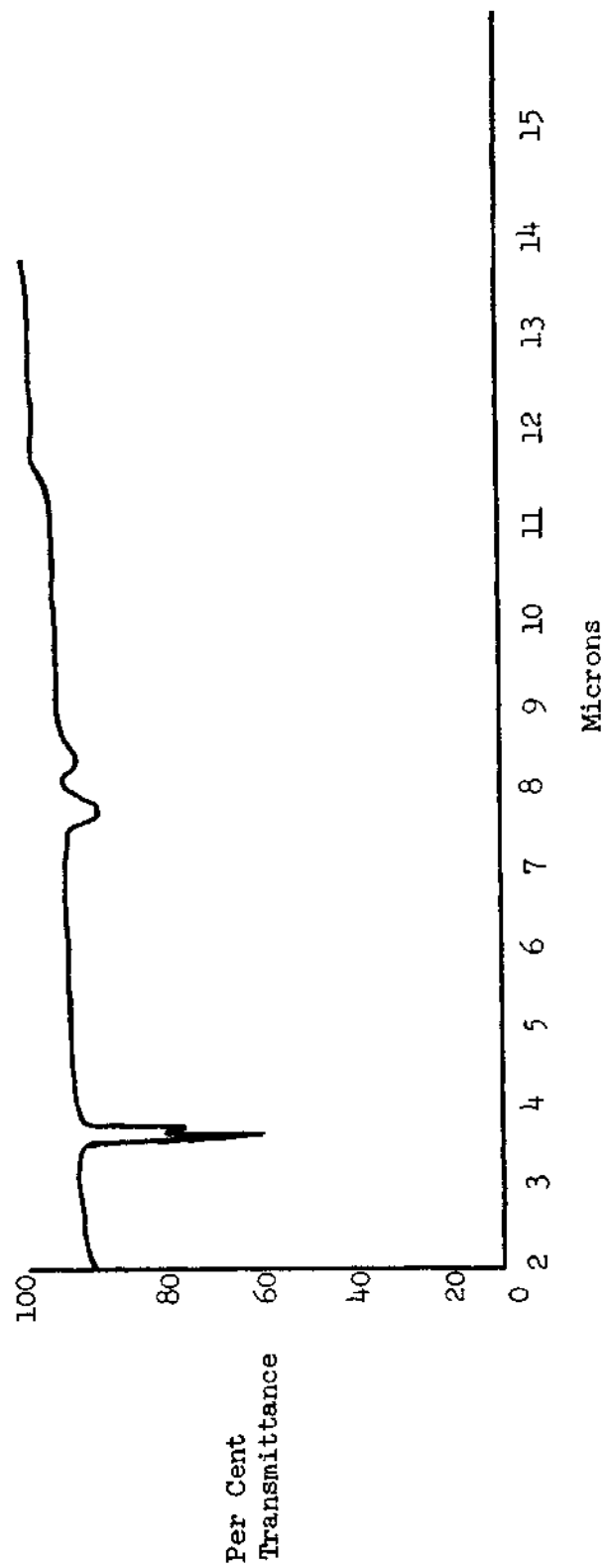


Figure 2. Characteristic Infrared Spectrum of Wash Solution Extract

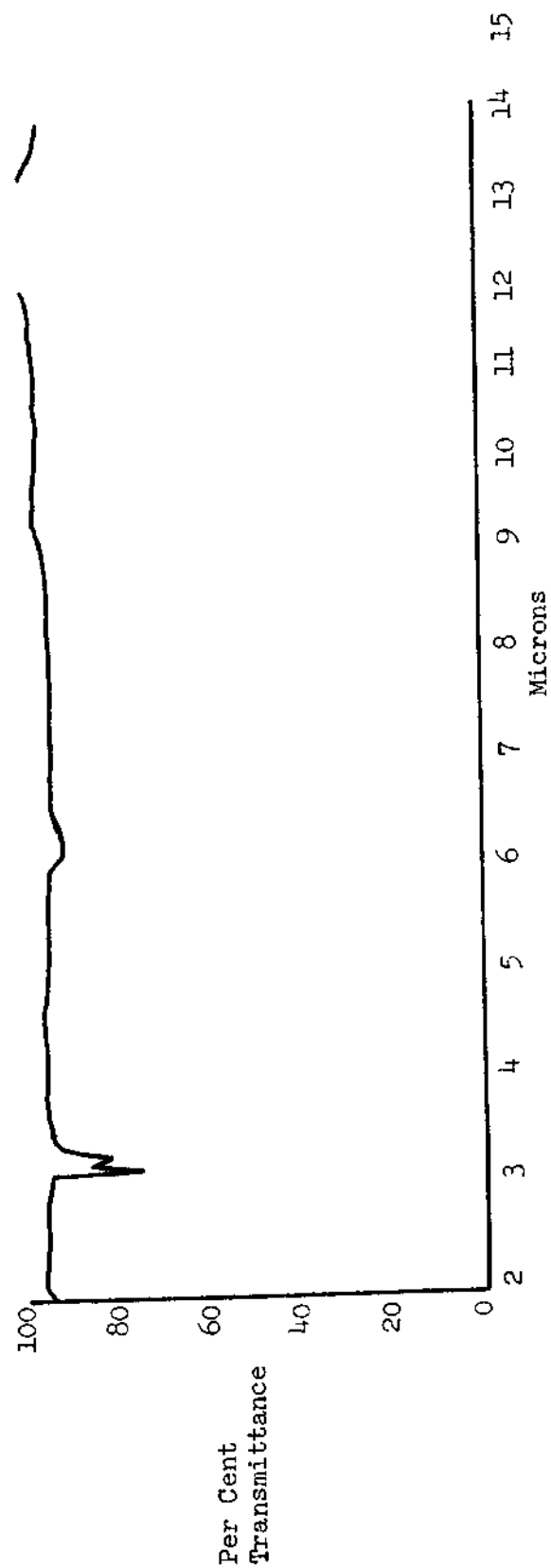


Figure 3. Characteristic Infrared Spectrum of Wash Solution Extract

Table 3. Desizing and Scouring Using Technical Grade Caustic, Recovered Caustic, and Enzymes

Sample	Per Cent Caustic		Per Cent Enzymes	Per Cent Non-lint Removed
	Recovered	Technical Grade		
1-A	6			14.64
1-B		6		14.21
1-C			2	12.50
2-A	4			13.33
2-B		4		13.26
2-C			2	10.18
3-A	2			9.59
3-B		2		8.95
3-C			2	7.36

scouring processes. This is the only criterion by which these processes can be adequately evaluated. The greater the non-lint removed, the better the appearance of the fabric.

#### Mercerizing

Luster determinations were made of fabric mercerized with technical grade and recovered caustic soda. The difference in reflection between the warp and filling of a sample is explained by the difference in the count of each of the yarns and the construction of the fabric. There was very little difference in the reflection of the light, see Table 4, from the surface of the fabrics mercerized with technical grade and recovered caustic soda. Figures 4 and 5 show two samples of luster determinations using the photoelectric cell. No visible difference could be detected between the mercerized samples.

Much of the fabric mercerized is frequently dyed. Convincing the dyer that proper shades can be obtained after mercerizing with recovered caustic soda may be the major concern to some. The results obtained from this study show that if the recovered caustic is treated in the simple fashions described in the experimental section, then very satisfactory products can be made. As stated previously, very few people can detect a difference in reflectance of 3 per cent, while 5 per cent is acceptable in many instances. According to the spectrophotometric curves made of the dyed fabrics in this study, those mercerized with recovered caustic differed less than 2.5 per cent, and in some cases were equivalent with those dyed fabrics mercerized with technical grade caustic, see Figures 6, 7, and 8. Figure 6 represents a light color where small differences

Table 4. Luster Determination for Samples Mercerized  
Using Recovered and Technical Grade Caustic  
Soda

Sample	Source of Caustic	Average Units Reflected from Zero Line	
		Warp	Filling
1-A	Technical Grade	14.88	16.69
1-B	Recovered	15.06	16.00
2-A	Technical Grade	12.91	15.13
2-B	Recovered	12.54	14.41
3-A	Technical Grade	12.69	14.75
3-B	Recovered	12.06	14.16
4-A	Technical Grade	8.22	10.19
4-B	Recovered	8.13	10.06

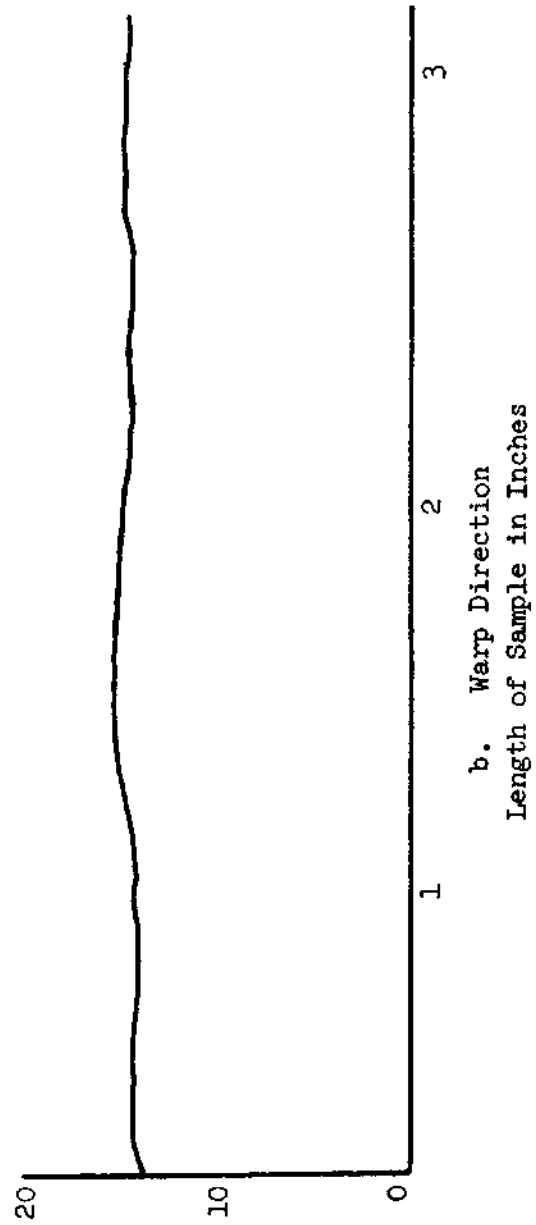
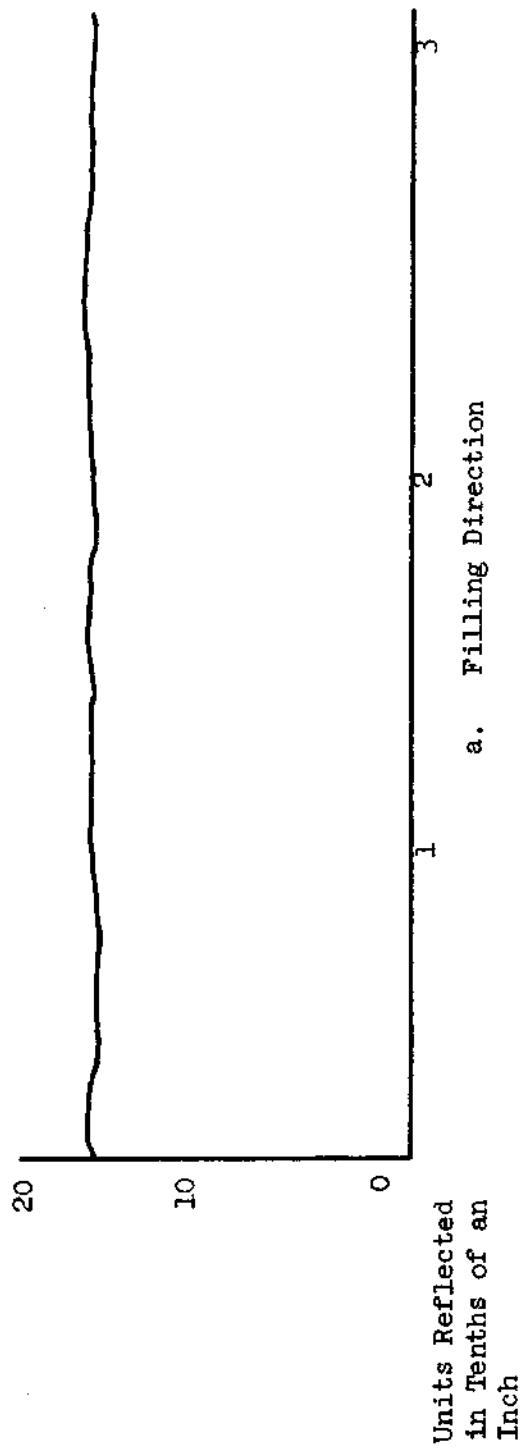


Figure 4. Luster Determination of Mercerized Fabric

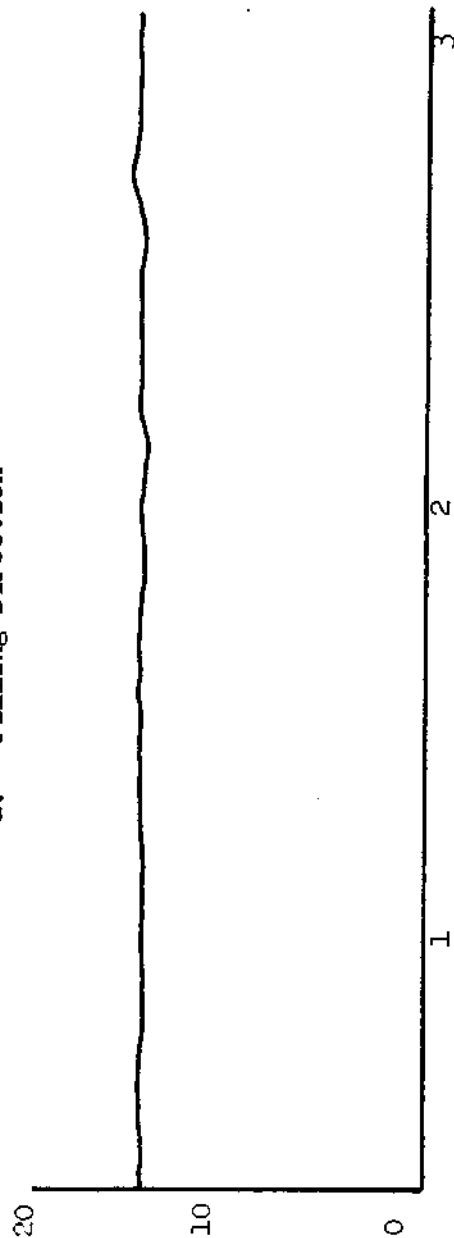
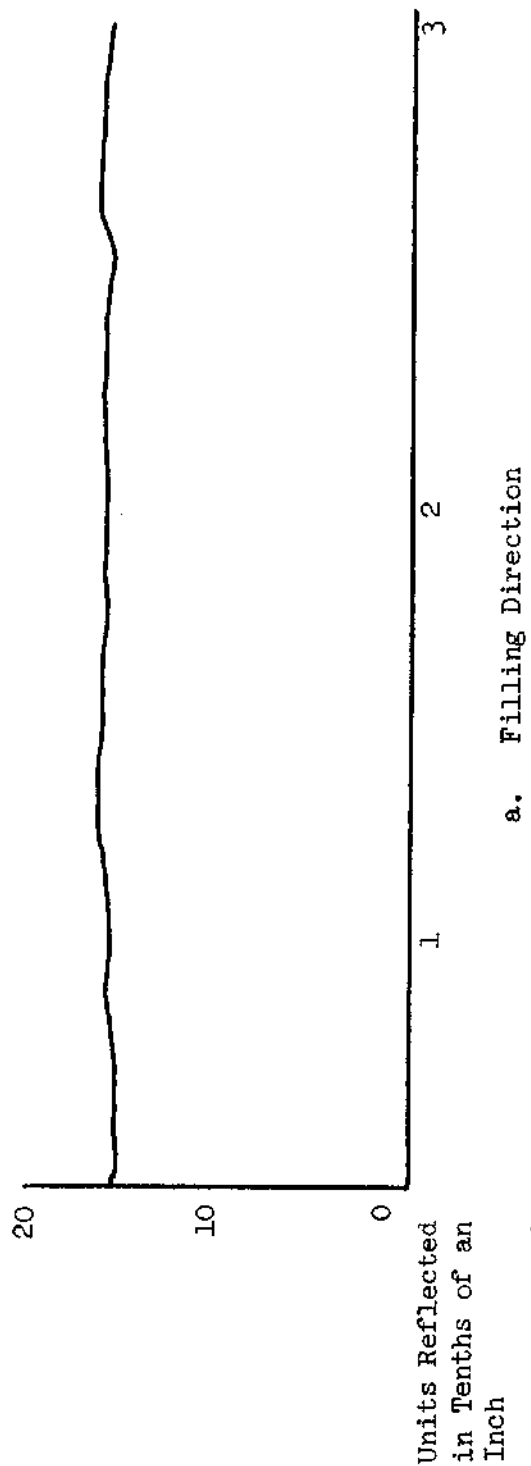


Figure 5. Luster Determination of Mercerized Fabric

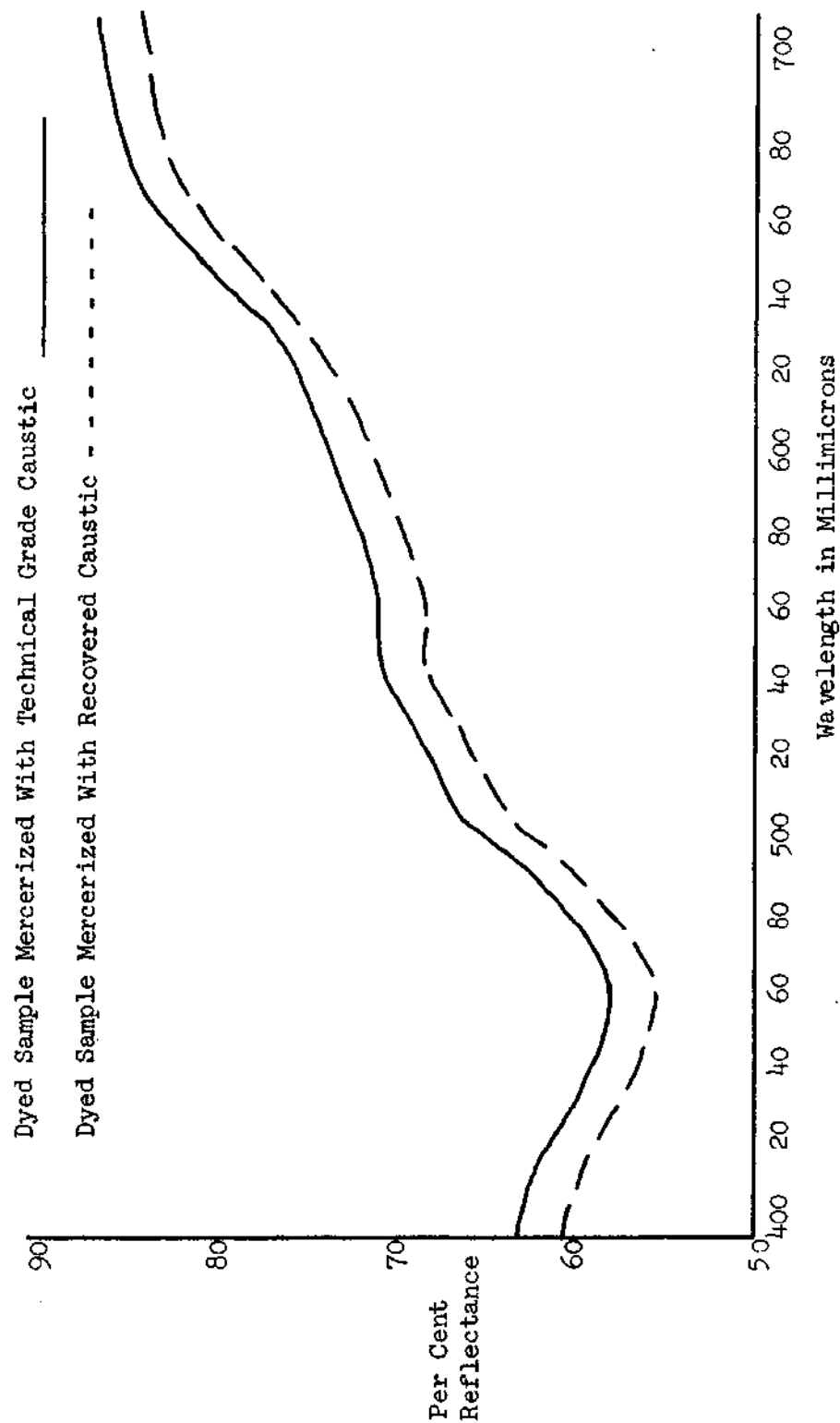


Figure 6. Comparison of Spectrophotometric Curves of Dyed Samples



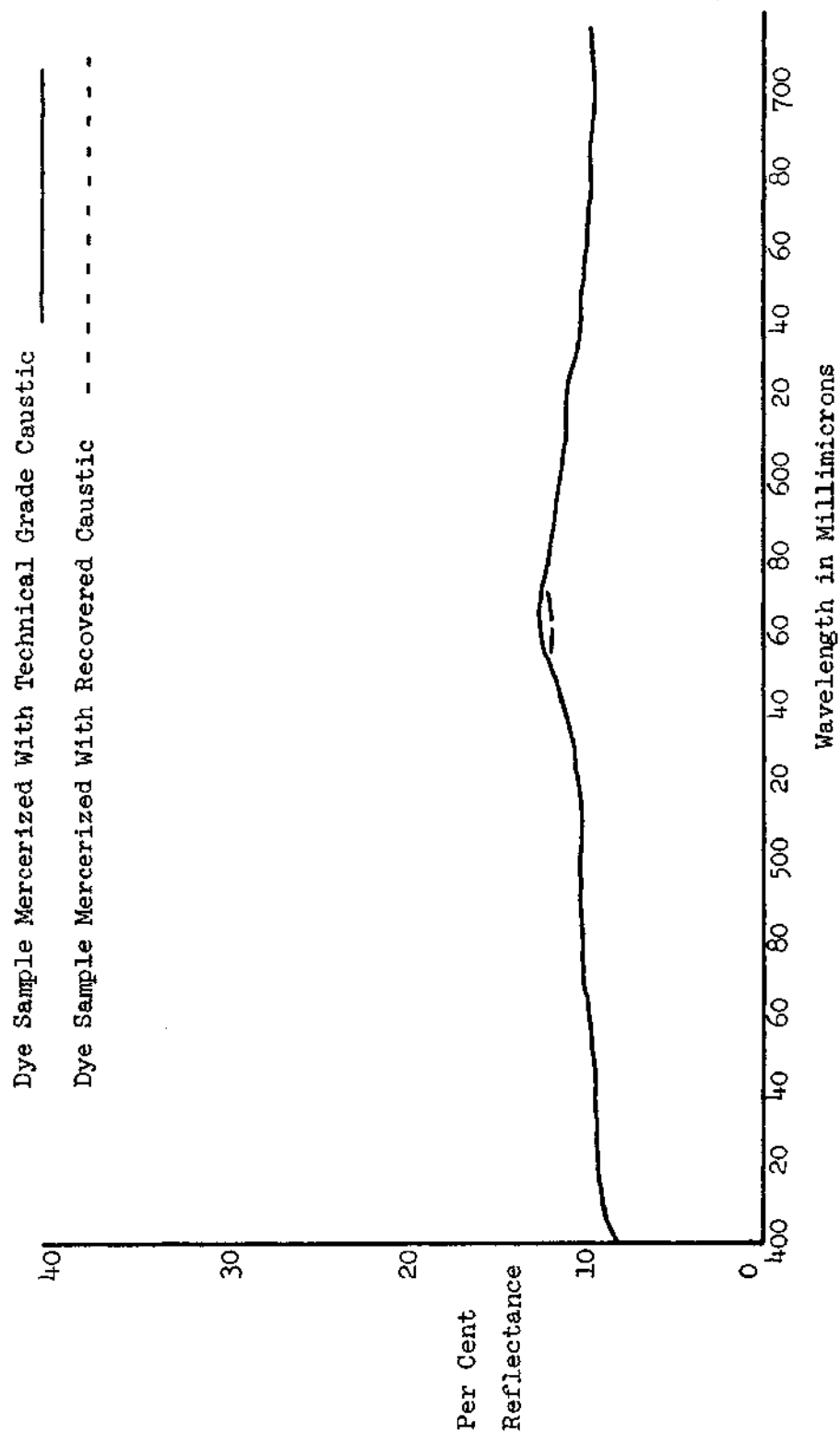


Figure 7. Comparison of Spectrophotometric Curves of Dyed Samples

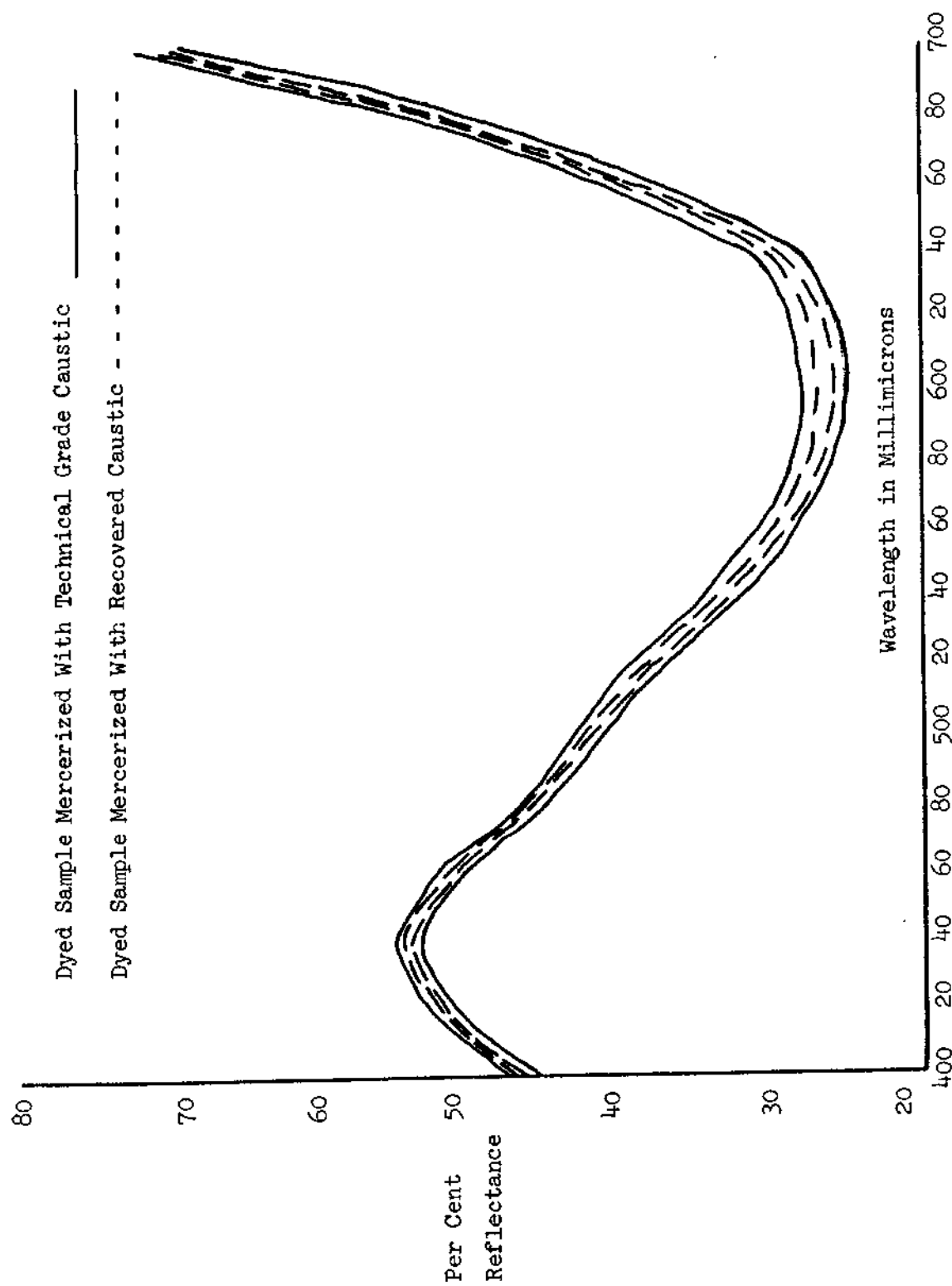


Figure 8. Comparison of Spectrophotometric Curves of Dyed Samples

Table 5. Dye Adsorption as a Function of Exposure Time During Mercerization with 22.5 Per Cent Caustic Soda

Sample	Time in Seconds	Units Reflected	Per Cent Absorbency
1*	15	100	100
2	30	84	118
3	45	80	125
4	60	80.8	124
5	105	84	118

Table 6. Dye Adsorption as a Function of Caustic Soda Concentration During Mercerization Using a Time Exposure of 75 Seconds

Sample	Concentration in Per Cent	Units Reflected	Per Cent Absorbency
6*	18	100	100
7	20	134.5	74.2
8	23	91.8	109

Note: Samples 1 and 6 were used as reference samples, and the units reflected were set at 100 with the following values expressed as a per cent of these samples.

are more pronounced than in Figure 7 which represents a dark color. Figure 8 represents fabrics with a light blue color. At all points on the curves, the two fabrics mercerized with recovered caustic soda were within the limits established by the two fabrics mercerized with technical grade caustic soda. No visible differences could be detected between any of the dyed samples.

#### Mercerization as a Variable in Dye Absorption

A light color gives a greater reflectance than a dark one. The lower values indicate that more dye has been absorbed by that particular sample than was absorbed by a corresponding sample of higher value. The per cent absorbancy is an inverse relationship of the light reflected. The results of Tables 5 and 6 indicate that the optimum mercerizing conditions for obtaining maximum dye absorption would be a caustic soda concentration of 23 per cent with the contact time between the cloth and caustic being 45 seconds. These values seem to be in close accordance with those found in the literature. This was true for both purified recovered caustic and technical grade caustic. There was no detectable difference between the two.

#### Bleach Manufacture

Jones' (12) work showed that if a satisfactory bleach could be made from recovered caustic right in the plant, a considerable savings could be made. Accordingly, as discussed in the experimental section, bleach solutions were made by adding chlorine directly to the purified recovered caustic. The resulting product was used at laboratory scale to determine its potency.

The strength of the bleach solution was continually checked during preparation, and the reaction was terminated when the bleach reached a strength of 7.85 grams per liter of available chlorine. The only strength requirement was that the bleach be as strong or stronger than that customarily used in industry. Depending upon the process employed, a bleach solution of 2 to 7 grams per liter of available chlorine may be used for 100 per cent cotton fabrics.

The bleach manufacturers use only reagent grade caustic soda in their processes because impurities decrease the stability of the bleach which may be stored for weeks before being sold to a customer. The grams per liter of available chlorine for the experimental bleach dropped from 7.85 to 7.41 over a five-day test period. This corresponds to a decrease of 5.61 per cent or a stability of 94.39 per cent. Thus, when a plant uses bleach of its own manufacture from recovered caustic and chlorine, it should be used within a five-day period.

The results of laboratory use of the experimental bleach for improving whiteness are compared to a commercial bleach in Table 7 with very little difference being detected.

Table 7. Comparison of Reflectance Using an Experimental and a Commercial Bleach

Sample	Strength of Bleach in Grams per Liter of Available Chlorine	Source	Average Units Reflected	
			Warp	Filling
1-A	2.0	Experimental	8.5	8.1
1-B	2.0	Commercial	8.3	8.2
2-A	2.0	Experimental	6.3	6.6
2-B	2.0	Commercial	6.5	6.2
3-A	3.0	Experimental	7.9	8.1
3-B	3.0	Commercial	7.8	7.9
4-A	3.0	Experimental	6.4	6.5
4-B	3.0	Commercial	6.4	6.6

## CHAPTER IV

### CONCLUSIONS AND RECOMMENDATIONS

#### Conclusions

The recovery and reuse of caustic soda from the mercerization process has previously been an economical advantage enjoyed only by those companies that could justify the purchase of sophisticated equipment. This investigation indicates that all plants, regardless of volume, which employ the mercerization process can feasibly recover and reuse caustic soda. Not only will this be economical, but it will reduce considerably the quantity of discarded caustic which pollute streams. Relatively few changes in the present equipment will be needed to install a satisfactory recovery system. Counter-current flow offers the best approach to the problem of recovering a caustic solution of appropriate concentration for reuse. If the counter-current flow method is not employed, as was the situation in this study, a "buffer" box can be used with good results.

The recovered caustic solution contained a wide range of solids which should be removed. The larger and heavier solids will settle out upon storage, while most of the smaller solids can be removed using a vibrating filter.

The analysis of the recovered caustic solution showed that there were no extraneous materials present that interfere with the satisfactory use of the recovered caustic in the processes studied. In fact, the recovered caustic contained constituents which were already present to some

degree in most of the solutions needed in the various wet processing.

Recovered caustic soda used in the selected textile processes gave very favorable results. Recovered caustic soda used for desizing and scouring gave better non-lint removal than the enzyme used and as good removal as the technical grade caustic.

The mercerization process is the largest user of caustic and will be capable of consuming most of the recovered caustic. The luster determinations made using recovered caustic soda equaled those recorded using technical grade caustic soda, and the subsequent dyeing of the mercerized fabrics showed that the recovered caustic had no detrimental effects on the spectrophotometric curves. Incidentally, these studies showed that by using optimum mercerizing conditions, a savings on the purchase of dyestuffs may also be realized with either recovered or technical grade caustic.

The experimental sodium hypochlorite prepared from recovered caustic proved to be strong enough and stable enough for use in bleaching. In many plants, hydrogen peroxide is used as the bleaching agent. Because of this, the use of the low cost experimental sodium hypochlorite for the treatment of textile effluents should be investigated as a possible attractive use.

Recovered caustic soda can be advantageously substituted for technical grade caustic soda in many processes. Regardless of whether an evaporator or a "buffer" box is used, caustic recovery and reuse can be profitable.



### Recommendations

This study was primarily concerned with the recovery and reuse of sodium hydroxide. This one chemical probably contributes more alkalinity to streams than any other in cotton textile finishing. Its removal from the streams will importantly contribute to a much reduced alkalinity and sodium ion content in the water supply. However, this is only one facet of the pollution problem from textile wastes. Much is still to be done by the textile industry to reduce water pollution. Some recommendations are:

- a. Caustic soda should be recovered and reused for mercerization, scouring, and as an alkaline agent to control pH because of (1) the savings realized in the form of more profits or a lower cost of production, and (2) the reduction in pollution.
- b. Recovered caustic plus chlorine to give a bleach or alkaline oxidizing agent should be investigated for treatment of effluents to remove color, odor, or other obnoxious conditions.
- c. The scope of this work was necessarily limited to caustic soda because of time. However, the entire range of materials discharged from textile operations deserve study, as suggested by Jones, for an entirely closed system to conserve water and chemicals and to avoid pollution.

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Note: The thesis by L. D. Jones gives a complete industry-wide bibliography. Also, an annotated but as yet unpublished bibliography, "State of the Art of Water Use and Waste Disposal in the Textile Industry," by L. D. Jones and W. L. Hyden, gives a comprehensive literature summary for 1950-1966.